

TEA/N/12 100 gm ea 25% NaOH

ETAZ (Final) = 252 ml

HPLC - TFA = 99.07 Imp = 0.38

700N E + 60N AC BF₃ = 140 gm 0 to 26°C

Temp raised to 25°C 2 ACA = 100 gm TFA/N/12 = 252 ml

Temp rose to 40°C stirring at 40 ± 2°C for 1 hr 30 min

HPLC

1 hr

1 hr 20 min

Workup - 500W DMW cooked 2m dropped 2m hydro pH adjusted to 3.50

at 25°C NH₃ = 185 ml stirred for one hr filtered washed Mat. with

yellow washing - 50 ml spray, 100 ml slurry, 100 ml spray

ETAZ - 50 ml spray 100 ml slurry

Dried by air 116.37

HPLC

2 ACA = 0.37

Fu = 97.1

Imp =

Fenaca/N/13

TFA/N/13 50 gm ea 25% extra NaOH (used old one)

EL₁ = 12% imp

EL₂ =

200 gm E + 300 ml AC + BF₃ 68.5 gm

TFA/N/13 = m temp 30°C

stirring